

Enforcement Analytical Method for Phosphorous Acid Technical

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| TITLE: Volumetric Determination of Phosphite | PAGE: 1 OF 6 |
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| CHANGES FROM PREVIOUS EDITION: Update of Critical Equipment List to Critical Equipment Spreadsheet (§ 6.1, 6.7, 12.6). Reorganization of references. | |

1. Purpose

This procedure describes an analytical method for determining the inorganic phosphite content of materials. Phosphite is oxidized to phosphate with iodine and the excess iodine back titrated with sodium thiosulfate.

2. Scope

This method applies to the analysis of water-soluble inorganic phosphites.

3. Definitions

3.1. The following abbreviations are used in calculations:

- 3.1.1. "W_s" is the mass of the sample in grams.
- 3.1.2. "V_T" is the volume of titrant in ml required to reach the end point in the titration of the sample.
- 3.1.3. "V_b" is the volume of titrant in ml required to reach the end point in the titration of the blank.
- 3.1.4. "N" is the normality of the sodium thiosulfate titrant, found in the TARGET NORMALITY column of the appropriate Titrant Standardization Schedule and on the label of the reservoir.

3.2. This procedure contains no other special terms requiring definitions.

4. Precision

4.1. The precision of this method was determined in Study CHB-EX-003 and reported in CHB-EX-003-005-P. In that study, seven replicate determinations were performed on a sample of 70 % phosphorous acid solution using this procedure, with the following results:

- 4.1.1. Mean Phosphorous Acid Content = 70.36 %.
- 4.1.2. Standard Deviation = 0.134 %.

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4.2. Errors may result if the normality of the sodium thiosulfate solution has changed since the last standardization. The titer should be verified at least every four weeks according to CHB-OP-CAL-004-P.

4.3. Reducing agents represent sources of interference with the chemistry of this method.

5. Safety

Normal laboratory safety practices should be observed.

6. Equipment

- 6.1. Analytical balance, appearing on the "Critical Equipment Spreadsheet".
- 6.2. Weighing dishes or weighing paper.
- 6.3. Erlenmeyer flasks, 250-ml capacity.
- 6.4. Stoppers to fit 250-ml flask.
- 6.5. Magnetic stir plate.
- 6.6. Magnetic stirring bar.
- 6.7. pH meter, appearing on the "Critical Equipment Spreadsheet".
- 6.8. Combination pH electrode comprising a glass measuring electrode and a silver/silver chloride reference electrode.
- 6.9. Burets, 50-ml, borosilicate glass, manufactured according to ASTM E287 Class A specifications.
- 6.10. Graduated cylinder, 25 ml.
- 6.11. Pipet, glass, 40-ml, Class A.
- 6.12. Pipet bulb.

7. Chemicals and Reagents

- 7.1. Deionized water, according to CHB-SP-CHE-001-L.
- 7.2. Sodium hydroxide, 50 % solution.
- 7.3. Sodium hydroxide, 0.3 N, according to CHB-SP-REA-004-P.
- 7.4. Sulfuric acid, 9 M, according to CHB-SP-REA-007-P.

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- 7.5. Sulfuric acid, 0.5 N, according to CHB-SP-REA-002-P.
- 7.6. Phosphate Buffer, pH 7.3, according to CHB-SP-REA-019-P.
- 7.7. Iodine, 0.1 N, purchased solution.
- 7.8. Acetic acid, glacial, ACS Reagent grade.
- 7.9. Sodium thiosulfate, 0.1 N, prepared according to CHB-SP-REA-005-P.
- 7.10. Starch indicator solution, according to CHB-SP-REA-006-P.

8. Calibration and Standardization

- 8.1. Standardization and verification are described in CHB-OP-CAL-004-P.
- 8.2. Use the TARGET NORMALITY from the Titrant Standardization Schedule for 0.1 N sodium thiosulfate.

9. Procedure

- 9.1. Weigh an amount of sample containing up to 0.03 g phosphite (as phosphorus) into a flask and record the weight W_s to the nearest 0.0001 g.
- 9.2. Add 80 ml deionized water and a stirring bar to the flask. Stir for one hour, or until the sample is dissolved.
- 9.3. Adjust the pH of the sample solution to 7.3 by dropwise addition of acid or base.
 - 9.3.1. Make coarse adjustments in pH by adding strong acid or strong base, as necessary.
 - 9.3.2. Make fine adjustments in pH by adding acidic titrant or basic titrant, as necessary.
 - 9.3.3. Rinse the pH electrode into the flask when pH adjustment is complete.
- 9.4. Prepare a blank containing 80 ml deionized water. Perform all subsequent steps on both the blank and the sample solution.
- 9.5. Add 25 ml pH 7.3 buffer. The same bottle of buffer must be used for both the sample and the blank. A graduated cylinder may be used.

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- 9.6. Add 40.0 ml iodine solution with gentle stirring. The same bottle of iodine must be used for both the sample and the blank. Either a buret or pipet may be used.
- 9.7. Stopper the flask and place in a dark location for 30 - 40 minutes.
- 9.8. Add 3 ml of glacial acetic acid.
- 9.9. Titrate with 0.1 N sodium thiosulfate to a pale straw color.
- 9.10. Add sufficient starch indicator to produce a dark navy blue color.
- 9.11. Continue the titration to a clear, colorless end point. Interpolate the buret reading to the nearest 0.05 ml and record the volume V_T of titrant used.
- 9.12. Repeat using a larger amount of sample if V_T is more than 35 ml, or a smaller sample if V_T is less than 5 ml.
- 9.13. Perform a blank titration by repeating Steps 9.5 through 9.11, and record the volume V_B of titrant used.

10. Calculations

- 10.1. Calculate the phosphorus(III) concentration as:

$$\% \text{ Phosphorus(III)} = \% P = (V_B - V_T) * N * 1.5487 / W_S$$

- 10.2. Calculate the phosphorus(III) concentration as phosphorous acid (H_3PO_3) or phosphorus pentoxide (P_2O_5), if required:

$$\% H_3PO_3 = \% P * 2.6474$$

$$\% P_2O_5 = \% P * 2.2913$$

11. Reporting

Report the percent phosphorus(III) or other calculated concentrations to the nearest 0.01 %.

12. References

- 12.1. "Laboratory Glass Graduated Burets", ASTM E287-94, American Society for Testing and Materials, West Conshohocken, PA (1994).
- 12.2. "Titrant Standardization Schedule", CHB-OP-CAL-002-S.

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- 12.3. "Standardization of 0.1 N Sodium Thiosulfate Solution", CHB-OP-CAL-004-P.
- 12.4. "Determination of the pH of Solutions", CHB-OP-MTH-001-P.
- 12.5. "Specifications for Deionized Water", CHB-SP-CHE-001-L.
- 12.6. "Critical Equipment Spreadsheet", CHB-SP-MS-009-S.
- 12.7. "Preparation of 0.5 N Sulfuric Acid Titrant", CHB-SP-REA-002-P.
- 12.8. "Preparation of 0.3 N Sodium Hydroxide Titrant", CHB-SP-REA-004-P.
- 12.9. "Preparation of Starch Indicator Solution", CHB-SP-REA-006-P.
- 12.10. "Preparation of Dilute Acid Reagents", CHB-SP-REA-007-P.
- 12.11. "Preparation of pH 7.3 Buffer", CHB-SP-REA-019-P.

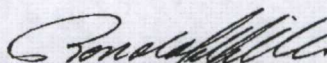
13. Attachments

None.

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14. Approvals

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